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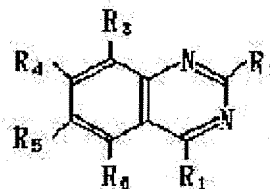
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(54) DEVELOPER COMPOSITION FOR BLACK AND WHITE SILVER HALIDE PHOTOGRAPHIC SENSITIVE MATERIAL

(57)Abstract:

PURPOSE: To provide the stable developer compsn. for black and white silver halide photographic sensitive materials which does not generate silver staining and is excellent in rapid processability without impairing fixability.

CONSTITUTION: The compd. expressed by the following formula is incorporated into the developer for black and white silver halide photographic sensitive materials. In the formula, R1 to R6 respectively denote hydrogen atoms, -SM1 groups, hydroxy groups, lower alkoxyl groups, -COOM2 groups, amino groups, or lower alkyl groups contg. -SO2M3 groups; at least one among R1 to R6 denote -SM1 groups. M1, M3, M3 respectively denote hydrogen atoms, alkaline metal atoms which may be the same or different.



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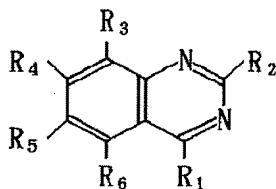
CLAIMS

[Claim(s)]

[Claim 1] The developer constituent for monochrome silver halide photosensitive material characterized by containing at least one sort of compounds expressed with the following general formula [1].

[Formula 1]

一般式 [1]



(In a general formula [1], R1-R6 are a hydrogen atom, one -SM, hydroxyl, a lower alkoxy group, two -COOM(s), the amino group, -SO3M3 set, or a low-grade alkyl group respectively, and at least one of R1-R6 shows one -SM.) M1, M2, and M3 express a hydrogen atom, an alkali-metal atom, or ammonium respectively, may be the same or may differ.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] About the new developer constituent for monochrome silver halide photosensitive material, this invention relates to the developer constituent for monochrome silver halide photosensitive material which silver dirt does not produce, even when especially rapid development processing is carried out.

[0002]

[Description of the Prior Art] Generally photographic processing of the monochrome silver halide photosensitive material is carried out in four processes of development, fixing, rinsing, and desiccation after imagewise exposure. Although processed with the developer with which hydroquinone, phenidone, or Metol was constructed and united, since development is performed in alkali, in order that many of developments may prevent oxidation of a developing agent and it may raise shelf life, a sulfite usually contains it in a developer. However, since this sulfite has the property to dissolve silver salt, silver salt is dissolved during a development out of photosensitive material, it is returned, and the silver salt which began to melt into the developer serves as metal silver, will deposit, and will adhere on the surface of sensitive material, and silver dirt will produce it. In the elevated-temperature quick processing especially using a conveyance mold auto-processor, silver dirt poses a problem.

[0003] Moreover, a problem is also serious, when there are few amounts of developer supplements and deposit concentration increases relatively to the throughput of sensitive material. In order to solve this problem, the present condition is not yet found out although research of a compound which raises shelf life, without dissolving silver salt is done. The research on the means which carries out the trap of the silver salt which dissolves on the other hand, and prevents a deposit has also been made. For example, retrieval of a silver sludge inhibitor is performed widely and 2-mercapto Benz imidazole sulfonic-acid derivative is reported [derivative / 1-phenyl-5-mercapto tetrazole] by U.S. Pat. No. 3,173,789 by JP,62-4702,B about the disulfide compound at JP,52-36029,A. However, by the approach using these, reduced the development rate and it was desensitized, and this developer was carried into the fixer of the following step, it reacted by the mothball of a developer and the new problem of that operation effectiveness disappearing was caused [**** / reducing a fixing rate].

[0004]

[Problem(s) to be Solved by the Invention] To the above problems, silver dirt is not generated but the technical problem of this invention is to offer the developer constituent for monochrome silver halide photosensitive material which moreover does not produce desensitization.

[0005] Other purposes of this invention are to offer the developer constituent for monochrome silver halide photosensitive material which does not spoil fixable. Another purpose of this invention is to excel in quick processability and offer the stable developer constituent for monochrome silver halide photosensitive material moreover.

[0006]

[Means for Solving the Problem] The above-mentioned technical problem of this invention is attained by [of the compound shown in the developer for monochrome silver halide photosensitive material by said general formula [1] (** 1)] making a kind contain at least.

[0007] Hereafter, it explains still more concretely about this invention.

[0008] It is the radical which they may have a substituent further and has 1-3 carbon numbers preferably, and in a general formula [1], the low-grade alkyl group and lower alkoxy group which are expressed with R1-R6 are a radical which has 1-5 carbon, respectively, and it is [the amino group

expressed with R1-R6 expresses a permutation or the unsubstituted amino group, and] a low-grade alkyl group as a desirable substituent. As ammonium, it is a permutation or unsubstituted ammonium, and is desirable unsubstituted ammonium.

[0009] As for R1-R6, it is desirable that hydroxyl, -COOM2, the amino group, and -SO3M3 set are included. At least one of R1-R6 is one -SM.

[0010] As the amount used, 10-5 to ten - one mol per 1l. of developers and further 10-4 to 10-2 are desirable. As pH of a developer, 9.5-12.0 are desirable, and the amount of developer supplements is 75 - 200 ml/m2 preferably two or less 200 ml/m.

[0011] Dry toDry of the processing time is 30 - 60 seconds preferably for 20 to 60 seconds.

[0012] Although the example of the compound shown by the general formula [1] below is shown, this invention is not limited to these.

[0013]

[Formula 2]

一般式〔1〕で表される化合物の具体例。

	R ₁	R ₂	R ₃	R ₄	R ₅	R ₆
1-1	-SH	H	H	H	H	H
1-2	-SH	-OH	H	H	H	H
1-3	-SH	H	H	H	H	-SO ₃ H
1-4	-SH	-CH ₃	-OH	H	H	H
1-5	-SH	-NH ₂	H	H	-OH	H
1-6	-SH	H	H	-NH ₂	H	-COOH
1-7	-SH	H	-CH ₃	-CH ₃	H	H
1-8	-SH	H	H	-SH	H	H
1-9	-SH	-OH	H	-SH	H	-C ₂ H ₅
1-10	-SH	H	H	-COOH	H	H
1-11	-OH	-SH	H	H	H	H
1-12	-SH	-SH	H	H	-OH	H
1-13	H	-SH	-OH	H	-CH ₃	H
1-14	H	-SH	-NH ₂	H	H	H
1-15	H	-SH	-OH	-CH ₃	H	H
1-16	H	-SH	-NH ₂	-C ₂ H ₅	-SH	H
1-17	H	-SH	H	-CH ₃	H	H
1-18	OH	-SH	H	H	H	H
1-19	H	-SH	H	-H	H	-COOH
1-20	H	-SH	H	-SO ₃ H	-SH	H
1-21	H	H	-SH	H	-OH	H
1-22	-OH	H	-SH	H	H	H
1-23	-OH	-CH ₃	-SH	H	H	H
1-24	-NH ₂	H	-SH	H	H	-C ₂ H ₅
1-25	-SH	H	-SH	H	H	-COOH
1-26	H	H	H	-SH	H	H
1-27	H	-OH	H	-SH	H	H
1-28	-OH	H	H	-SH	H	H
1-29	-NH ₂	H	H	-SH	H	H
1-30	H	-NH ₂	H	-SH	H	H
1-31	H	-NH ₂	-CH ₃	-SH	H	H
1-32	-SH	H	H	H	-SH	H
1-33	-SH	-CH ₃	H	H	-SH	H
1-34	H	-OCH ₃	H	H	-SH	H
1-35	-SH	-SH	H	H	-SH	H
1-36	H	-CH ₃	-CH ₃	H	-SH	H
1-37	-H	-NH ₂	H	H	H	-SH
1-38	-SO ₃ H	-SH	H	H	H	-SH
1-39	-H	-OH	-SH	-C ₂ H ₅	H	-SH
1-40	-H	-H	H	H	H	-SH
1-41	-H	-SH	OH	H	-CH ₃	-SH
1-42	-H	-H	H	-SH	H	-SH

[0014] The compound of this invention is a well-known compound, and can come to hand easily.

[0015] The compound of this invention has the work which the trap of the dissolved silver is carried out [work] and does not generate silver dirt on sensitive material, and the silver dirt prevention effectiveness of a developer is made to maintain, and the effectiveness excellent in mothball nature is shown. Therefore, a quick development becomes possible and the fall of a fixing rate can be prevented.

[0016] As a developing agent which can be used in this invention dihydroxybenzene (for example, hydroquinone and KURORU hydroquinone --) Prome hydroquinone, 2, 3-dichloro hydroquinone, methyl hydroquinone, Isopropyl hydroquinone 2, 5-dimethyl hydroquinone, etc., 3-pyrazolidone (for example, 1-phenyl-3-pyrazolidone and 1-phenyl-4-methyl-3-pyrazolidone --) The 1-phenyl -4, 4-dimethyl-3-pyrazolidone, 1-phenyl-4-ethyl-3-villa ZORIDON, aminophenols (for example, ortho aminophenol --), such as 1-phenyl-5-methyl-3-pyrazolidone Para aminophenol, N-methyl-ortho aminophenol, N-methyl-para aminophenol, Pyrogallol, such as 2 and 4-diaminophenol, an ascorbic acid, 1-aryl-3-pyrazolines (for example, 1-(p-hydroxyphenyl)-3-amino pyrazoline --) 1-(p-methylamino phenyl)-3-amino pyrazoline, 1-(p-aminophenyl)-3-amino pyrazoline, 1-(p-amino-N-methylphenyl)-3-amino pyrazoline, etc. -- etc. -- independent -- or, although it can be combined and used It is desirable to use it in the combination of 3-pyrazolidone and dihydroxybenzene or the combination of aminophenols and dihydroxybenzene. As for a developing agent, it is desirable to usually be used in the amount of 0.01-1.4 mols/l.

[0017] In this invention, there are a sodium sulfite, potassium sulfite, ammonium sulfite, sodium metabisulfite, etc. as the sulfite and metabisulfite which are used as preservatives. I. of a sulfite is desirable in 0.25 mols /or more. It is 0.4 mols/l. or more especially preferably.

[0018] In addition to this in a developer, as occasion demands Alkali chemicals (a sodium hydroxide, potassium hydroxide, etc.), a buffer for pH (for example, a carbonate, phosphate, a borate, a boric acid, and an acetic acid --) dissolution assistants (for example, polyethylene glycols --), such as a citric acid and alkanolamine sensitizers (for example, the nonionic surface active agent containing polyoxyethylenes --), such as those ester and alkanolamine Surface active agents, such as the fourth class ammonium compound, a defoaming agent, a fogging inhibitor for example, the silver halide like a potassium bromide and a sodium bromide and nitro bends indazole -- Nitrobenzimidazole, benzotriazol, benzothiazole, chelating agents (for example, ethylenediaminetetraacetic acid or its alkali-metal salt --), such as tetrazoles and thiazoles accelerators (for example, U.S. Pat. No. 2,304,025 --), such as a nitrilotriacetic acid salt and a polyphosphate The compound of a publication etc. can add hardening agents (for example, a glutaraldehyde or its bisulfite addition product etc.) or a defoaming agent in JP,47-45541,B each official report. As for pH of a developer, being prepared by 9.5-12.0 is desirable.

[0019] The compound of this invention may be used for the activator processing liquid which a developing agent is included [liquid] in sensitive material, for example, an emulsion layer, and makes negatives develop by processing sensitive material in an alkali water solution as a special format of a development. Such a development is used as one of the approaches of quick processing of sensitive material combining the silver salt stabilizing treatment by the thiocyanate in many cases, and is also applicable to such processing liquid. In such quick processing, especially the effectiveness of this invention is large.

[0020] The thing of a presentation generally used as a fixer can be used. A fixer is a water solution which generally consists of a fixing agent and others, and pH is usually 3.8-5.8. What is known for the organosulfur compound which can generate fusibility stability silver complex salt besides thiocyanates, such as thiosulfates, such as a sodium thiosulfate, potassium thiosulfate, and ammonium thiosulfate, a sodium thiocyanate, a potassium thiocyanate, and ammonium thiocyanate, as a fixing agent as a fixing agent can be used.

[0021] The water-soluble aluminum salt which acts as a hardening agent, for example, an aluminum chloride, an aluminum sulfate, potash alum, etc. can be added to a fixer.

[0022] Compounds, such as preservatives (for example, a sulfite, a bisulfite), pH buffer agent (for example, acetic acid), and a chelating agent with pH regulator (for example, sulfuric acid) water-softening ability, can be included in a fixer by request.

[0023] The viscous liquid of the half-boiling condition that the organic nature water solution of viscosity containing mixture, or the glycol and amine of a fixed component is also expensive is sufficient as a developer. Moreover, at the time of use, it may dilute, and you may use, or may use as it is.

[0024] On the occasion of the development of this invention, development temperature can also be set as the 20-30-degree C usual temperature requirement, and it can also be set as the range of 30-40-degree C high temperature processing.

[0025] As for monochrome silver halide photosensitive material by this invention, being processed using an auto-processor is desirable. It is processed filling up the developer of the constant rate which is

proportional to the area of sensitive material on that occasion. The amount of development supplements is 250ml or less per two 1m, in order to lessen the amount of waste fluid. It is 75ml or more per two 200ml or less 1m preferably. In the amount of developer supplements of less than 75ml per two, the satisfactory photograph engine performance is not obtained by desensitization, bearish-ization, etc. 1m. [0026] As for this invention, it is desirable that all the processing times (Dry to Dry) when processing using an auto-processor from the request of developing time compaction, after a film edge is inserted in an auto-processor until it comes out from a desiccation zone are 20 - 60 seconds. All the processing times here are the time amount which is specifically need at processing including all process time amount required to process monochrome silver halide photosensitive material and which contained all of the time amount of processes, such as development, fixing, bleaching, rinsing, stabilizing treatment, and desiccation, for example, i.e., the time amount of Dry to Dry. In less than 20 seconds, the photograph engine performance with all the processing times satisfactory at desensitization, bearish-izing, etc. is not obtained. All the processing times (Dry to Dry) are 30 - 60 seconds still more preferably.

[0027]

[Example] Next, this invention is not limited by these examples although an example explains this invention to a detail.

[0028] After carrying out corona discharge to the polyethylene terephthalate which carried out example 1 under-coating processing with the energy of 8W/(m² and min), the antistatic liquid of the following configuration was applied with the speed of 30 m/min using the roll fit coating pan and the Ayr knife so that it might become the following adhesion volume.

[0029] (Preparation of a base material which has a conductive layer) After carrying out corona discharge to polyethylene terephthalate with a thickness of 100 micrometers which carried out under-coating processing, the antistatic liquid of the following configuration was applied with the speed of 70 m/min using the roll fit coating pan and the Ayr knife so that it might become the following adhesion volume.

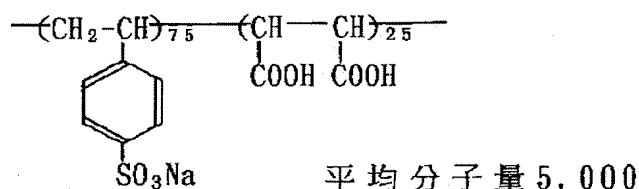
[0030]

Water-soluble conductive polymer P-6 0.6 g/m² Hydrophobic polymer particle L-1 0.4g/m² Polyethylene oxide compound Ao-1 0.06 g/m² Hardening agent E-8 0.2 g/m² 90 degrees C, it dried for 2 minutes and this was heat-treated for 90 seconds 140 degrees C. What applied this conductive layer to one side of a base material was prepared.

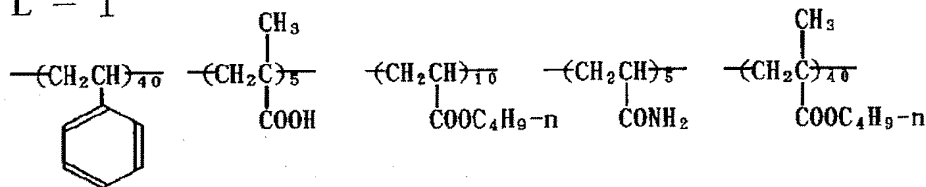
[0031]

[Formula 3]

P - 6



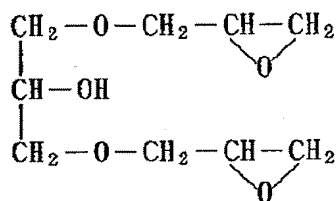
L - 1



{ A o - 1 }



E - 8



[0032] (Preparation of a silver halide emulsion) The salt iodine-silver-bromide (62 mol % and silver iodide-sized 0.5-mol % of silver chlorides etc. is silver bromide) emulsion was prepared using coincidence alligation.

[0033] In addition, hexa BUROMO rhodium acid potassium salt and 8xten - seven mols of hexa chloro iridium acid potassiums were added with 8xten - eight mols per one mol of silver, respectively at the time of a mixed process after 5% of the last attainment mean diameter is formed until it results in the last attainment mean diameter.

[0034] After desalting the obtained emulsion by the usual flocculation method using the derivatized gelatin processed by phenyl isocyanate, it distributed in gelatin, and the emulsion which adds the following [A], [B], and [C] as an antibacterial, and consists of a cube monodisperse particle (10% of coefficient of variation) with a mean particle diameter of 0.30 micrometers was obtained.

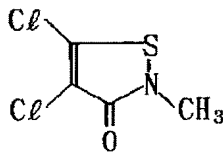
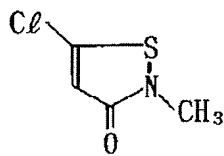
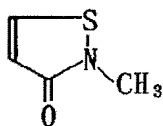
[0035]

[Formula 4]

{ A }

{ B }

{ C }



[0036] After adding a citric acid, a sodium chloride, and 1-phenyl-5-mercapto tetrazole to this emulsion, adding chloroauric acid and a sodium thiosulfate, carrying out chemical ripening at 60 degrees C and reaching the highest sensibility, it is 4-hydroxy-6-methyl. - After adding 1, 3, 3a, and 1g per one mol of silver of 7-TETORAZA indenenes and stopping aging, 600mg of potassium bromides per one mol of silver halides and 150mg of sensitizing dye of following structure SD-1 were added.

[0037] (Preparation of emulsion coating liquid) this emulsion -- per one mol of silver halides -- the

sodium salt of 2 and 4-dichloro-6-hydroxy-1,3,5-triazine was added as a saponin and a hardening agent, having used [hydroquinone / inhibitor ST-1] a sodium-hydroxide solution (2g and 1N) and the following structure S-1 as 1.5g and a spreading assistant for 150mg and a styrene-maleic-acid polymer having used the polymeric latex P1 of 4g and the following structure as 15g.

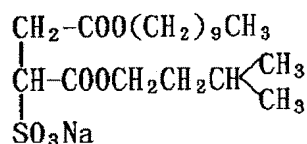
[0038] (Preparation of emulsion protective coat layer coating liquid) The following structure S-2 and a citric acid were added having used [the formalin addition product of sodium bisulfite] 15mg of mono dispersion silicas with 5.5mg, a mean particle diameter [of 3 micrometers], and a mean particle diameter of 8 micrometers at a time as the spreading assistant for 1mg and 1-phenyl-4-hydroxymethyl-3-pyrazolidone at the water solution which contains per two and gelatin 1.1g 1m, respectively, and formalin was further added as a hardening agent. Moreover, surfactant FA-33 of a fluorine system were added so that coverage might be set to 3xten - six mols/m².

[0039] (Preparation of backing layer coating liquid) In the water solution which contains per two and gelatin 2.3g 1m after-mentioned water-soluble-dye compound III-1 -- 100mg -- said -- III-2 25mg said -- for III-3 100mg and polymeric latex P-1 350mg 60mg and colloidal silica were added as the mixture of 150mg, [A], [B], and [C], and a spreading assistant, glyoxal and E-2 [55mg] were added as sodium dodecylbenzenesulfonate and a hardening agent, and the styrene-maleic-acid polymer was agitated.

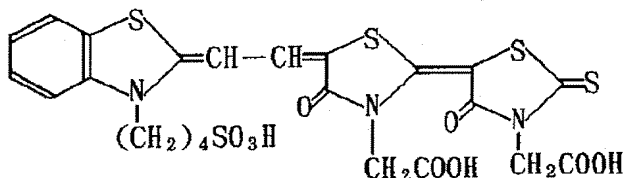
[0040]

[Formula 5]

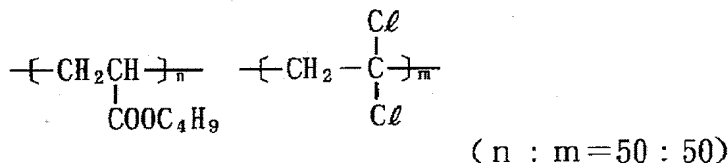
S - 2



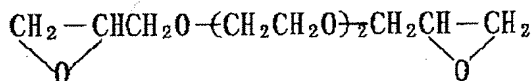
S D - 1



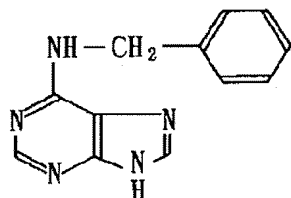
P - 1



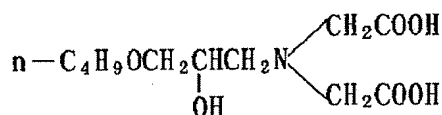
E - 2



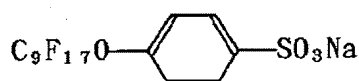
S T - 1



S - 1



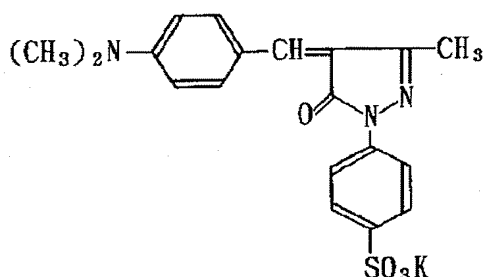
F A - 33



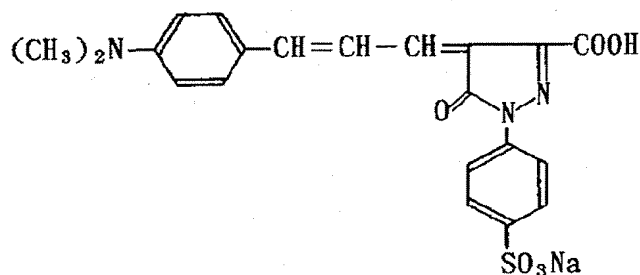
[0041]

[Formula 6]

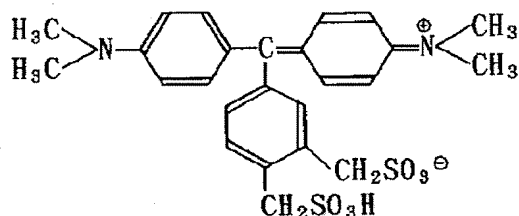
化合物 III - 1



化合物 III - 2



化合物 III - 3



[0042] (Preparation of backing layer protective coat layer coating liquid) The mixture of 7mg, the distributed object of mono dispersion polymethylmethacrylate of 5.5 micrometers of mean diameters, [A], [B], and [C] and a styrene-maleic-acid polymer were added in the water solution which contains per two and gelatin 0.7g 1m, S-2 was agitated in it, and the sodium salt of glyoxal and 2, and 4-dichloro-6-hydroxy-1,3,5-triazine was further added as a hardening agent.

[0043] (Creation of a sample) After carrying out corona discharge to one side on the polyethylene terephthalate film base material which has the above-mentioned antistatic layer with the energy of 15W/(m² and min), the backing layer coating liquid and backing layer protective coat layer coating liquid which were prepared as mentioned above to the side which has the above-mentioned antistatic layer were applied. Moreover, after carrying out corona discharge to one side on a base material with the energy of 15W/(m² and min), the emulsion layer and the emulsion protective coat layer were applied. In addition, it applied and the emulsion layer was dried so that it might become silver content 4.0 mg/m² and amount of gelatin 1.7 mg/m².

[0044] Subsequently, the development was carried out on the following conditions in platemaking auto-processor GQ and 26SR (Konica make) using a following developer and a following fixer.

[0045] The film was processed without exposing.

[0046]

<Processing conditions> [developer formula]

A disodium ethylenediaminetetraacetate salt 2g Diethylene glycol 25g Potassium sulfite (55% W/V water solution) 110ml Potassium carbonate 55g Hydroquinone 20g 5-methyl benzotriazol 200mg The compound of the general formula [1] of this invention, or comparison compound (shown in Table 1) 1.0xten - three mols Potassium hydroxide Amount which sets pH of a used solution to 10.5 Potassium bromide 5.0g 1-phenyl-3-pyrazolidone 750mg These were dissolved in pure water, and it finished and used for 1l.

[0047]

[Fixer formula]

((A) Presentation)

Ammonium thiosulfate (72.5% W/V water solution) 230ml Sodium sulfite 9.5g Sodium acetate and 3 monohydrate 28g Boric acid 6.7g A sodium citrate and 2 monohydrate 2g Acetic acid (90%W/W water solution) Amount which sets pH of a used solution to 4.7 (presentation B)

Pure water 17ml Sulfuric acid (50% W/W water solution) 2.5g Aluminum sulfate (an aluminum₂O₃ conversion content is a W/W water solution 8.1%) 21g It melted in order of the above-mentioned presentation A and the presentation B in 500ml of water at the time of use of a fixer, and finished and used for 1l.

[0048] [Development conditions]

Process Temperature Time amount development 38 degrees C 12-second fixing 35 degrees C 10-second rinsing Ordinary temperature 10-second desiccation 50 degrees C 13-second sum total Each process time amount also includes the so-called rear-spring-supporter conveyance time amount to degree process for 45 seconds.

[0049] The replenisher used the same thing as the presentation of a developer and a fixer, and it processed the sample two times 30m, supplying with 190 cc of development 160 cc/m² fixing/, and the rate of m². In order to investigate the existence of the silver dirt after processing, the 3.5x12cm piece of a film was processed with unexposed, and visual observation of the dirt on the front face of a film was carried out. Evaluation of development dirt was considered as five-step evaluation, what has the severest dirt was considered as the rank 1, and the thing without dirt was considered as the rank 5. It is in a condition practically nonpermissible in less than three rank.

[0050] Development rate: It expressed with the relative sensibility when carrying out a development on said conditions, after exposing for 10 to 6 seconds by helium-Ne laser.

[0051] Fixing rate: The time amount (second) from which a sensitive-material sample becomes transparence with the fixer when processing 2 30m was measured, and it expressed with relative velocity.

[0052] Shelf life: The prepared developer was put in polyethylene wrapping and heat-treatment was carried out 60 degrees C for 20 days. After heat-treatment, the silver stain test was performed and five-step evaluation was performed. The thing with the severest dirt was considered as the rank 1, and the thing without dirt was considered as the rank 5. It means that the prevention effectiveness of silver dirt has not disappeared even if a developer is heated so that it is close to a rank 5. It is in a condition practically nonpermissible in less than three rank.

[0053] The result is shown in Table 1.

[0054]

[Table 1]

試験	現像液中の化合物	現像汚れ	現像速度	定着速度	保存性	備考
1	—	1	100	100	1	比較
2	比較化合物 a	3	60	45	2	"
3	比較化合物 b	4	65	100	2	"
4	比較化合物 c	4.5	73	94	2	"
5	化合物 1-1	4.5	95	95	5	本発明
6	" 1-3	5	100	98	5	"
7	" 1-11	5	100	100	5	"
8	" 1-18	5	100	100	5	"
9	" 1-20	5	100	100	5	"
10	" 1-23	4.5	98	98	5	"
11	" 1-28	4.5	98	98	4	"
12	" 1-32	5	94	95	5	"
13	" 1-38	5	98	100	5	"

a : 1-フェニル-5-メルカプトテトラゾール

b : 2-メルカプトベンズイミダゾール

c : ビスフェニル酢酸-2-ジスルフィド

[0055] The shelf life of a developer was also found by that the trials 5-22 using the developer which added the compound of this invention are good, without there being almost no development dirt and

reducing a development rate and a fixing rate so that clearly from Table 1.

[0056]

[Effect of the Invention] Silver dirt was able to be improved by this invention, and it was able to excel in the shelf life of a developer, fixable was not able to be spoiled, but the developer constituent which can be processed quick was able to be offered.

[Translation done.]